PB84-187889

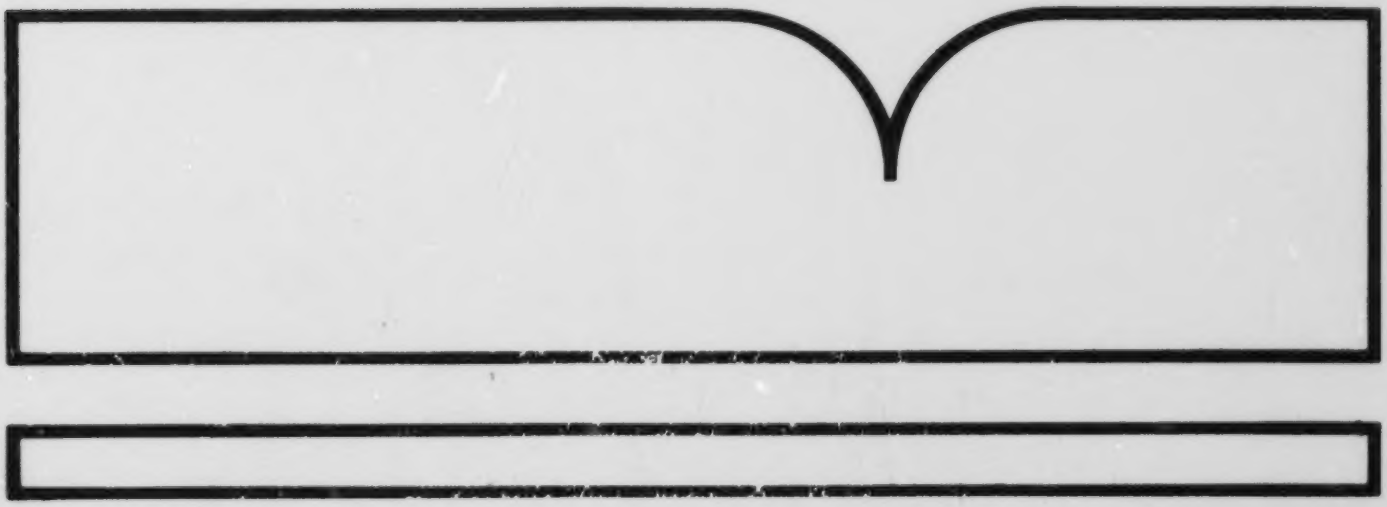
Performance Audit Results for POHC (Principle Organic Hazardous Constituents) VOST (Volatile Organic Sampling Train) and Bag Measurement Methods

Research Triangle Inst.
Research Triangle Park, NC

Prepared for

Environmental Monitoring and Support Lab.
Research Triangle Park, NC

May 84



U.S. Department of Commerce
National Technical Information Service
NTIS

PB84-187889

EPA-600/4-84-036
May 1984

PERFORMANCE AUDIT RESULTS FOR POHC: Vost and Bag Measurement Methods

by

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EPA Contract 68-02-3431

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SPRINGFIELD, VA. 22161

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/4-84-036	2.
3. RECIPIENT'S ACCESSION NO. PB84 187889	
4. TITLE AND SUBTITLE: Performance Audit Results for POHC: VOST and Bag Measurement Methods	5. REPORT DATE: May 1984
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S): R.K.M. Jayanty, J.A. Sokash, W.F. Gutknecht and C.E. Decker, Research Triangle Institute	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Research Triangle Institute, Research Triangle Park, NC 27709	10. PROGRAM ELEMENT NO. DU-D109, Project-B1401
	11. CONTRACT/GRANT NO. Contract No. 68-02-3431
12. SPONSORING AGENCY NAME AND ADDRESS: U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Research Triangle Park, NC 27711	13. TYPE OF REPORT AND PERIOD COVERED: Final report
	14. SPONSORING AGENCY CODE: EPA/600/08

15. SUPPLEMENTARY NOTES

16. ABSTRACT

This report includes the results of a pilot audit program on source emission measurement systems used to measure POHC (Principle Organic Hazardous Constituents). Four laboratories currently active in sampling and analysis of POHC during trial burns at hazardous waste incenerators were included in the pilot audit program. Two source emission measurement systems frequently used for volatile POHC measurement during trial burns were audited. These measurement systems are the volatile organic sampling train (VOST) and bag measurement technique. The audits were conducted using gas cylinders containing five organics (carbon tetrachloride, chlorform, perchoroethylene, vinyl chloride and benzene). Cylinder concentrations of 7 to 90 ppb for each organic was used for the VOST audits and concentrations of 90 to 430 ppb was used for the bag measurement audits. Results from the pilot audit program demonstrate gas cylinders are a very effective means of assessing accuracy of volatile POHC measurement systems. Audit results are reported in the report.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
VOST, POHC, audits, carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, benzene, hazardous waste incineration		

18. DISTRIBUTION STATEMENT: Release Unlimited	19. SECURITY CLASS *(This Report)*: Unclassified	21. NO. OF PAGES: 41
	20. SECURITY CLASS *(This page)*: Unclassified	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

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1.0 INTRODUCTION

Under the regulations of the Resource Conservation and Recovery Act (RCRA) of 1976, the Administrator of the U.S. Environmental Protection Agency (EPA) has promulgated performance standards for owners and operators of hazardous waste treatment, storage, and disposal facilities. The RCRA regulations, which deal specifically with hazardous waste incineration, are described in the Code of Federal Regulations, Title 40, Parts 122, 264 and 265. Hazardous waste incinerators are tightly regulated under the Act. However, burning of hazardous waste materials in industrial boilers and cement manufacturing rotary kilns is excluded from the RCRA regulations under "Energy Exempt" status. Therefore, EPA has recently initiated a test program to determine the destruction/removal efficiency (DRE) for principle organic hazardous constituents (POHC) in industrial boilers and rotary kilns so that the RCRA regulations may be extended in the future to these types of operations. The determination of DRE requires a reliable sampling and analytical method which may be used with confidence during the hazardous waste trial test burns. EPA is currently recommending a Volatile Organic Sampling Train (VOST) method for collection and analysis of volatile (bp 30-100°C) POHC emissions from hazardous waste incinerators (see Ref. 1).

Although the VOST is intended for compounds with bp 30-100°C, it appears the method is applicable to compounds outside this range. In particular, of the compounds selected for the audits, vinyl chloride has a bp of -14°C and perchloroethylene has a bp of 121°C.

Research Triangle Institute (RTI) was directed by the Quality Assurance Division of EPA's Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, to conduct performance audits to evaluate the accuracy and precision of the sampling and analysis phases of the VOST method. RTI used a multicomponent mixture of five organics in compressed gas cylinders as an audit material to evaluate the VOST method at four laboratories. All four laboratories have active programs for POHC measurements using the VOST method either as contractors to EPA or to other organizations. As an alternative to the VOST method for volatile POHC, the accuracy and precision of sampling and analysis using a bag technique was also evaluated. This report summarizes the objectives of the audit program, the

audit procedures used and the laboratories audited, the performance audit results, and pertinent conclusions and recommendations.

2.0 OBJECTIVES

The main objectives of this audit program are: (1) to demonstrate that cylinder gases can be used to assess and document the accuracy and precision of volatile POHC measurements during trial burns of hazardous waste, (2) to screen the sampling and analysis phases of the VOST and bag methods to determine the principal sources of measurement error, (3) to evaluate the proficiency of laboratories currently using the VOST or bag methods for POHC measurements, and (4) to determine the stability of the POHC at parts-per-billion (ppb) concentrations in gas cylinders.

3.0 AUDIT PROCEDURES

3.1 Preparation of Audit Material and Verification

Gas cylinders containing a mixture of five organic compounds in a balance gas of nitrogen were used as the audit material for this study. The five compounds in the audit cylinders were: carbon tetrachloride (tetrachloromethane), chloroform, perchloroethylene (tetrachloroethylene), vinyl chloride, and benzene. The audit cylinders contained compounds in two concentration ranges. A low concentration cylinder in the range of 7 to 90 ppb for each POHC was used for the evaluation of the VOST method. A high concentration cylinder in the range of 90 to 430 ppb for each POHC was used for the evaluation of the bag measurement method. The audit cylinders were prepared and analyzed by the National Bureau of Standards (NBS) under an Interagency Agreement with EPA. The NBS values for the low and high concentration audit cylinders were as follows:

	Concentration (ppb)	
Compound	Low	High
Carbon tetrachloride	21	141
Chloroform	23	120
Perchloroethylene	29	126
Vinyl chloride	31	154
Benzene	18	133

After NBS analysis, the audit cylinders were sent to the Environmental Sciences and Engineering Unit at RTI. RTI analyzed the cylinders before and after conducting the VOST/bag audits to demonstrate the stability of the organic compounds during the audit period. RTI analysis results were in close agreement with the NBS-measured concentrations (see Table 1, Section 4). In addition, the NBS and RTI analyses showed the organic compounds to be stable over the entire study period (approximately 4 months).

3.2 Audit Description and Laboratories Audited

Four laboratories currently involved in POHC measurements were the subjects of performance audits. The laboratories were: Battelle Columbus Laboratories; Pedco Environmental, Inc.; Radian Corporation (formerly TRW Environmental Engineering Division); and Engineering Science, Inc. (sampling was performed by Engineering Science and GC/MS analysis was performed by the Analytical and Chemical Sciences Unit at RTI). All four laboratories participated in the VOST audits but only two laboratories were involved in the bag audits. All four laboratories were given the names of the organic compounds in the audit cylinder, which was used for the VOST method and the concentration range of each compound (i.e., 7 to 90 ppb). All four laboratories were audited with the same 7 to 90 ppb VOST audit cylinder. The two laboratories participating in the bag audits were told the names of each compound and the concentration range of each compound (i.e., 90 to 430 ppb). Both laboratories were audited with the same 90 to 430 ppb audit cylinder. The proposed audit sequence and general instructions for the sampling and analysis of volatile POHC from quality assurance (QA) audit cylinders were sent to each laboratory prior to the actual audit. A copy of the audit sequence and general instructions provided to the laboratories is shown in Appendix A. Audits were conducted to evaluate both the sampling and analysis phases and the analysis phase only of the VOST/bag methods. The audits were conducted by R. K. M. Jayanty and J. A. Sokash, RTI, at the four laboratory sites. During each audit, the measurement procedures were initially reviewed with the appropriate laboratory staff. The sampling and analysis procedures used by the respective laboratories are summarized in Appendix B.

4.0 SUMMARY OF RESULTS

4.1 Volatile Organic Sampling Train (VOST)

Audits were conducted at four laboratories to evaluate the combined sampling and analysis accuracy and precision of the VOST method. In addition, accuracy and precision of the analytical component was also evaluated. As per Instruction 3 (see Appendix A), each laboratory collected three pairs of samples using the entire VOST system and three pairs of samples on VOST organic sorption cartridges only from the VOST audit gas cylinder. The cartridge only configuration was used to simulate the analytical component of the VOST method. All samples were collected from the audit cylinder at a flow rate of 0.5 L/min for 20 min for a total volume of approximately 10 L. The collected samples were analyzed by three of the laboratories (Laboratories A, B and D) using thermal desorption purge and trap and gas chromatography/mass spectrometry (GC/MS). One laboratory (Laboratory C), however, analyzed the collected samples by gas chromatography using PID/Hall detectors since they plan to use this GC technique for the determination of POHC in the field. Laboratory C analyzed only three organics (carbon tetrachloride, chloroform and benzene). All four laboratories analyzed at least one pair of VOST (Tenax-Tenax/charcoal) cartridges as blanks. After the analyses of the audit samples, the results were submitted to RTI and the EPA Project Officer for further evaluation.

A summary of all VOST audit results including laboratory accuracy is shown in Table 1. A summary of laboratory precision for VOST audit results is presented in Table 2 (see Appendix C). The analysis results reported by the laboratories for each individual measurement of the five compounds and their percent accuracy relative to the expected values are tabulated in Tables 3-7 (see Appendix C). The percent accuracy for each compound was calculated using the NBS-determined concentrations as the true values. A summary of the accuracy results for the three laboratories using GC/MS is presented on Page 6.

TABLE 1. SUMMARY OF AUDIT RESULTS

Activity Audited	Audit Gases	Audit Gas True Conc. and Stability (1)(2)(5) NBS Conc. ppb	RTI 10/83 ppb	RTI 12/83 ppb	Lab A (6) Range (ppb)	Lab A (5) Average ppb	Lab A (5) Average %(3) Acc.	Lab B Range (ppb)	Lab B (5) Average ppb	Lab B (5) Average %(3) Acc.	Lab C (4) Range (ppb)	Lab C (5) Average ppb	Lab C (5) Average %(3) Acc.	Lab D (6) Range (ppb)	Lab D (5) Average ppb	Lab D (5) Average %(3) Acc.
VOST (Both sampling & analysis)	carbon tetrachloride	21	21.7	21.1	5.9-15.3	10.4	-50	24.5-30.4	26.6	27	22.0-24.0	23.0	10	15.0-21.0	18.0	-14
	chloroform	23	23.8	23.2	21.7-25.2	23.1	0	28.8-29.8	29.4	28	26.0-30.0	27.7	20	18.0-19.0	18.5	-20
	perchloroethylene	29	30.4	31.1	29.7-36.3	33.9	17	35.4-41.4	38.8	34	-----NOT AUDITED-----			29.0-36.0	32.5	12
	vinyl chloride	31	34.9	28.0	12.8-18.2	15.5	-50	7.8-13.0	10.5	-66	-----NOT AUDITED-----			24.0-32.0	28.0	-10
	benzene	18	19.4	20.0	22.9-24.1	23.5	31	22.9-24.8	23.7	32	15.0-17.0	16.3	-9	19.0-19.0	19.0	6
VOST Cartridges analysis only	carbon tetrachloride	21	21.7	21.1	10.4-14.9	12.7	-40	22.6-24.0	23.3	11	22.0-27.0	23.7	13	19.0-20.0	19.5	-7
	chloroform	23	23.8	23.2	23.6-25.0	24.3	6	25.8-27.0	26.4	15	26.0-38.0	30.3	32	18.0-19.0	18.5	-20
	perchloroethylene	29	30.4	31.1	37.5-38.5	38.0	31	30.9-35.8	33.4	15	-----NOT AUDITED-----			33.0-36.0	34.5	19
	vinyl chloride	31	34.9	28.0	13.1-20.8	16.4	-47	7.7-21.4	14.6	-53	-----NOT AUDITED-----			32.0-39.0	35.5	15
	benzene	18	19.4	20.0	25.4-27.0	26.2	46	21.5-23.4	22.5	25	21.0-21.0	21.0	17	17.0-19.0	18.0	0
BAG (Both sampling & analysis)	carbon tetrachloride	141	148	149	222-230	225	60	121-124	123	-13						
	chloroform	120	117	117	160-163	161	34	99-103	101	-16						
	perchloroethylene	126	128	136	153-181	165	31	70-73	71	-44	-----NOT AUDITED-----			-----NOT AUDITED-----		
	vinyl chloride	154	161	158	86-126	104	-33	96-105	101	-34						
	benzene	133	136	143	147-167	157	18	90-101	97	-27						
Direct Gas Analysis by GC Only	carbon tetrachloride	141	148	149	223-252	238	69	131-135	133	-6						
	chloroform	120	117	117	169-176	173	44	115-117	116	-3						
	perchloroethylene	126	128	136	97-104	101	-20	116-126	121	-4	-----NOT AUDITED-----			-----NOT AUDITED-----		
	vinyl chloride	154	161	158	87-136	118	-23	101-107	104	-33						
	benzene	133	136	143	160-180	173	30	117-119	118	-11						

(1) NBS (Gas and Particulate Division) and RTI (Environmental Sciences and Engineering unit) values were obtained by direct GC analysis.

(2) RTI values are shown to illustrate cylinder gas stability.

(3) % accuracy = $\frac{\text{Lab average value - NBS value}}{\text{NBS value}} \times 100$

(4) Analysis by GC and not GC/MS.

(5) Each concentration for RTI and all the laboratory values are an average of two or three analyses.

(6) Analyses were performed 2 to 3 weeks after sample collection.

Audit Gas	Range of average percent accuracy using GC/MS	
	VOST (Entire Train)	VOST (Cartridges Only)
Carbon tetrachloride	-50 to 27	-40 to 23
Chloroform	-20 to 28	-20 to 15
Perchloroethylene	12 to 34	15 to 31
Vinyl chloride	-66 to -10	-53 to 15
Benzene	6 to 32	0 to 46

In general, the range of percent accuracy for the entire VOST was within the ±50 percent accuracy limits reported in the VOST protocol (see Ref. 1). The only exceptions are the vinyl chloride and carbon tetrachloride results from Laboratory A and vinyl chloride results from Laboratory B, which were outside the expected accuracy limits described in the VOST protocol. Similar ranges of percent accuracy were found for directly loaded VOST cartridge samples. Thus the audit results from the entire VOST method and the VOST cartridges alone indicate that the variations in the percent accuracy were due to analytical error and not sampling error. Blank concentrations for the five compounds from the Tenax and Tenax/charcoal cartridges were found to be <1.0 ppb under laboratory conditions (see Table 6, Appendix C). One laboratory (Laboratory B) analyzed the Tenax and Tenax/charcoal cartridges separately. This laboratory noted that four audit gases (carbon tetrachloride, chloroform, perchloroethylene and benzene) were collected principally on the Tenax cartridges (>90 percent). However, vinyl chloride was found principally on the Tenax/charcoal cartridge (see Table 7, Appendix C). Statistical analyses have not been performed on the VOST measurements at present. As the number of audit results increase, detailed statistical evaluations will be performed and results will be presented in a future report.

4.2 Bag Measurements

Audits were conducted at two laboratories (Laboratories A and B) to evaluate the combined sampling and analysis accuracy and precision for bag measurements. In addition, the accuracy and precision of the analytical component only was evaluated.

As per Instruction 1 (see Appendix A), each laboratory collected audit gas in the same bag three times for analysis. The two laboratories used different types of bags for collection of samples. Laboratory A used a Tedlar bag and Laboratory B used a five-layer bonded bag with polyethylene as the innermost layer (see Appendix B). Between each analysis the bag was evacuated, filled with dry nitrogen, and then analyzed to show the absence of POHC memory effects. Each bag sample was analyzed in triplicate by direct transfer of the sample from the bag into the gas chromatograph. Also, as per Instruction 2 (see Appendix A), the audit gas was analyzed three times directly from the audit cylinder. All the audit samples were analyzed by gas chromatography with flame ionization detection or photoionization detection for the analysis of benzene and vinyl chloride and electron capture detection for the analysis of carbon tetrachloride, chloroform and perchloroethylene. The triplicate analysis results reported by Laboratories A and B for the bag measurements and direct analyses are tabulated in Tables 8-10 (Appendix C). The percent accuracies for each analysis were calculated relative to the NBS concentrations and are also included in these tables. A summary of the percent accuracies for each compound for both bag measurement (sampling plus analysis) and direct analysis only are shown in Table 1. The ranges of percent accuracy obtained for each compound for the bag measurements and the direct analyses are as follows:

	Range of average percent accuracy using GC	
	bag measurements	direct analysis
Carbon tetrachloride	-13 to 60	- 6 to 69
Chloroform	-16 to 34	- 4 to 44
Perchloroethylene	-44 to 31	-20 to - 4
Vinyl chloride	-34 to -33	-33 to -23
Benzene	-27 to 18	-11 to 30

Statistical analyses have not been performed on the bag measurements results at present. As the number of audit results increases, detailed statistical evaluations will be performed and these results will be presented in a future report.

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based upon the audit program objectives stated in Section 2.0 and the audit results described in Section 4.0 and Tables 1 through 10, the following conclusions are made from this audit program:

1. Over the 4-month study period, the low ppb audit mixture of five organic compounds (carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene) was stable (see Table 1). This mixture of organic compounds would be a good audit material.

2. The use of cylinder gases for performance audits on VOST and bag methods was successful in assessing the accuracy and precision for the combined sampling and analysis of POHC. However, the audit gas should be collected in the VOST (or bag, as appropriate) in the presence of control agency personnel. In this way, direct analysis of the audit cylinder will not be attempted.

3. Four laboratories may not be representative of the type of results that currently could be expected from all laboratories conducting POHC measurements during trial burns. However, the audit results indicate that laboratories can analyze POHC with accuracy to within the limits of ±50 percent that are stated in the VOST protocol (see Ref. 1). All audit results were within ±50 percent accuracy for VOST measurements except Laboratory A results for vinyl chloride and carbon tetrachloride and Laboratory B results for vinyl chloride, which were slightly beyond these limits (see Table 1).

4. Screening the data from the sampling and analysis phases for the VOST and bag methods shows:

   a. The analysis phase and not sampling is the principal source of error (see Table 1) for the VOST method. For the bag method, the choice of bag materials can have a significant effect on results. However, the analysis phase appears to be the major source of error for the bag method (see Table 1).

   b. Direct analysis with GC rather than GC/MS may yield higher accuracy for field tests where the POHC of concern are not mixed with a large number of other organic compounds (see Table 1-Laboratory C versus Laboratories A, B, and D for VOST results).

   c. Blank concentrations for VOST cartridges were found to be less than 1 ppb for each of the five organics under laboratory conditions (see Table 6).

d. For the five organics studied, greater than 90 percent of the carbon tetrachloride, chloroform, perchloroethylene, and benzene were collected on the Tenax cartridge. However, greater than 90 percent of the vinyl chloride was collected on the Tenax/charcoal cartridge (see Table 7).

5.2 Recommendations

Compressed gas cylinders containing five organic compounds (carbon tetrachloride, chloroform, perchloroethylene, vinyl chloride, and benzene) are currently available from EPA for audits to evaluate the accuracy and precision of data collected using the VOST and bag methods. In addition, the EPA is now in the process of preparing gas cylinders containing eight other organic compounds at low parts-per-billion levels (15 to 150 ppb) for performance audits. These eight organic compounds are: trichloroethylene, 1,2-dichloroethane, 1,2-dibromoethane, acetonitrile, trichlorofluoromethane, dichlorodifluoromethane, bromomethane, and methylethylketone. It is recommended that a performance audit using EPA audit cylinders be conducted during each hazardous waste trial burn test to assess and document the accuracy and precision of POHC measurements. In addition, an audit may be useful prior to the trial burn to evaluate the proficiency of the source test organization for POHC measurements. Any Federal, State, or Local agency, or its contractor, planning hazardous waste trial burn tests for POHC may request a performance audit by contacting the EPA's Regional Quality Assurance Management Office or the EPA's Environmental Monitoring Systems Laboratory, Quality Assurance Division, Research Triangle Park, North Carolina.

6.0 REFERENCE

1. Protocol for the Collection and Analysis of Volatile POHC using VOST, August 1983. Copy available from Technical Support Office, Industrial Environmental Research Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

APPENDIX A
PROPOSED AUDIT SEQUENCE AND THE INSTRUCTIONS PROVIDED TO THE LABORATORIES FOR THE SAMPLING AND ANALYSIS OF VOLATILE POHC FROM QUALITY ASSURANCE AUDIT CYLINDERS

PROPOSED AUDIT SEQUENCE FOR THE SAMPLING AND ANALYSIS OF VOLATILE POHC FROM QUALITY ASSURANCE AUDIT CYLINDERS

I. AUDIT OF TEDLAR® BAG SAMPLING AND ANALYSIS PROCEDURES

Step 1. Using a 90-to 430-ppb audit cylinder, collect samples in three bags and analyze each bag three times as per written instructions (INSTRUCTION SET NO. 1). Report values for all analyses. THIS IS AN AUDIT OF BOTH THE SAMPLING AND ANALYSIS PROCEDURES.

Step 2. Using the same 90- to 430-ppb audit cylinder, analyze the audit cylinder gas contents directly three times. This can be accomplished by injecting the audit gas directly into the instrument. Report values for all analyses. THIS IS AN AUDIT OF THE ANALYSIS PROCEDURE ONLY.

II. AUDIT OF VOST SAMPLING AND ANALYSIS PROCEDURES

Step 3. Using a 7- to 90-ppb audit cylinder, pass 10 L of audit gas through Tenax® and Tenax®/charcoal VOST collection cartridges only as per written instructions (INSTRUCTION SET NO. 2). Perform analysis by thermal desorption, gas chromatography/mass spectrometry (GC/MS). Repeat the sample collection and analysis procedure two more times. Report all three analysis results. THIS IS AN AUDIT OF THE ANALYSIS PORTION ONLY FOR VOST.

Step 4. Using the same 7 to 90 ppb audit cylinder, pass 10 L of audit gas through the volatile organic sampling train (VOST) as per written instructions (INSTRUCTION SET NO. 3). Perform analyses by thermal desorption, GC/MS. Repeat the sample collection and analysis procedures using the VOST two more times. Report all three analysis results. THIS IS AN AUDIT OF THE COMBINED VOST SAMPLING AND ANALYSIS PROCEDURES.

INSTRUCTION SET NO. 1

INSTRUCTIONS FOR THE SAMPLING AND ANALYSIS OF VOLATILE POHC FROM QUALITY ASSURANCE AUDIT CYLINDERS USING THE BAG TECHNIQUE

1.0 PURPOSE

This audit is intended to assess and document the accuracy and precision of the principle organic hazardous constituents (POHC) using the bag technique during trial burns at hazardous waste burning facilities. This audit also can be used to determine the proficiency of source testing organization prior to the actual trial burn tests.

2.0 PRINCIPLE

A gas sample containing POHC is drawn from an audit gas cylinder into a bag. The collected POHC are analyzed by gas chromatography according to the method specifications and procedures.

3.0 AUDIT MATERIAL (GAS CYLINDERS)

The audit material is contained in gas cylinders. A maximum of 5 L of audit gas is to be used for each test run. A two-stage gas regulator, 1/4-inch male/female glass joints, and Teflon® tubing are supplied for use with the gas cylinder.

4.0 PROCEDURES

The following step-by-step procedures should be used for sample collection and analysis using the bag technique.

Step A. Evacuate the bag with a vacuum pump and fill the bag with approximately 5 L of organic-free nitrogen from a gas cylinder. Tedlar® bags are recommended. Determine the hazardous constituents of interest by gas chromatography. Report the concentrations determined as Blank Value 1. The blank value must be not more than twice the detection limit. If it is higher, the bag cleaning procedure should be repeated.

Step B. After the blank analysis, evacuate the bag with a vacuum pump and fill the bag with approximately 5 L of audit gas from the audit cylinder. The 1/4 inch male/female glass joints that are supplied should be used as connections to the bag and the audit cylinder. Minimum Teflon® tubing should be used. Determine the POHC of interest by gas chromatography. Report the concentrations of each constituent as Analysis Result 1.

Step C. Evacuate the bag contents and fill the bag with organic free nitrogen (Cleaning Step 1). After 3 minutes, evacuate the bag contents and fill the bag again with nitrogen (Cleaning Step 2). After three minutes, repeat the evacuation procedure and fill the bag with 5 L of nitrogen (Blank 2). Determine the concentration of the bag contents by gas chromatography and report the measured concentration as Blank Value 2. The blank value must not be more than twice the detection limit. If it is higher, the bag cleaning procedure should be repeated.

Step D. Evacuate the bag contents and fill the bag with approximately 5 L of audit gas from the audit cylinder. Determine the POHC of interest by gas chromatography. Report the concentrations determined as Analysis Result 2.

Step E. Repeat Step C and report the measured concentration of the bag contents as Blank Value 3. The blank value must not be more than twice the detection limit. If it is higher, the bag cleaning procedures should be repeated.

Step F. Evacuate the bag contents and fill the bag with approximately 5 L of audit gas from the audit cylinder. Determine the POHC of interest by gas chromatography. Report the concentrations determined as Analysis Result 3.

5.0 SAMPLE ANALYSIS

Follow the procedures described in the method used for sample analysis, calibration, and calculations. Submit the results to the Project Officer and to the audit supervisor for evaluation.

INSTRUCTIONS FOR THE DIRECT ANALYSIS OF VOLATILE POHC FROM QUALITY ASSURANCE AUDIT CYLINDERS USING VOST CARTRIDGES

1.0 PURPOSE

This audit is intended to assess and document the accuracy and precision of the gas chromatography/mass spectrometry (GC/MS) analysis procedure or equivalent method used during trial burns at hazardous waste burning facilities. This audit also can be used to determine the proficiency of the analyst prior to the actual trial burn tests.

2.0 PRINCIPLE

A gas sample containing principal organic hazardous constituents (POHC) from an audit gas cylinder is passed through a Tenax® cartridge and a Tenax®/charcoal cartridge at a known flow rate using the rotameter supplied. These cartridges are the same type used in a volatile organic sampling train (VOST). The collected POHC on the cartridges are analyzed by thermal desorption, GC/MS or an equivalent method approved by EPA.

3.0 AUDIT MATERIAL (GAS CYLINDERS)

The audit material is supplied in gas cylinders. A maximum of 10 L of gas is to be used for each test run. A two-stage gas regulator, a rotameter, 1/4 inch male/female glass joints and Teflon® tubing are supplied for use with the gas cylinder.

4.0 PROCEDURES

The gas sample from the audit cylinder is passed through the VOST cartridges at a flow rate of 0.5 L/min using the rotameter supplied. Male/female glass joints and Teflon® tubing, which are supplied, should be used as connections to the rotameter and VOST cartridges. The volume of sample for any pair of the traps should not exceed 10 L. The gas stream and the VOST cartridges are maintained at room temperature.

Repeat the sample collection two more times with new pairs of sample cartridges. After the sample collection in each run, remove the two VOST

cartridges, replace the end caps, and immediately perform the analysis using thermal desorption, GC/MS or an equivalent method approved by EPA.

5.0 SAMPLE ANALYSIS

Follow the procedures described in the EPA method used for sample analysis, calibration, and calculations. Report the amount of each constituent and blank values to the Project Officer and to the audit supervisor for evaluation.

INSTRUCTIONS FOR THE SAMPLING AND ANALYSIS OF VOLATILE POHC FROM QUALITY ASSURANCE AUDIT CYLINDERS USING THE VOLATILE ORGANIC SAMPLING TRAIN

1.0 PURPOSE

This audit is intended to assess and document the accuracy and precision of the volatile organic sampling train (VOST) when used during trial burns at hazardous waste burning facilities. This audit also can be used to determine the proficiency of source testing organization prior to the actual trial burn tests.

2.0 PRINCIPLE

A gas sample containing principal organic hazardous constituents (POHC) is passed from an audit gas cylinder into a glass manifold and is used in place of the test site stack sample. While the POHC gas is being passed into the glass manifold, a major portion of the gas is drawn through a VOST according to the method specifications and procedures. The collected POHC are analyzed by thermal desorption, purge and trap, and gas chromatography/mass spectrometry (GC/MS) or an equivalent method approved by the EPA.

3.0 APPARATUS

3.1 GLASS MANIFOLD

A glass manifold is supplied. One side of the glass manifold is connected with Teflon® tubing to the audit cylinder. The other end of the glass manifold should be connected with Teflon® tubing to a rotameter and the excess gas from the rotameter should be vented into a hood. The glass manifold is heated to 100°C with heating tape. The heating tape, rotameter, 1/4-inch male/female glass joints, and Teflon® tubing are also supplied. One port of the glass manifold should be connected to the VOST probe with 1/4-inch male/female glass joints. A schematic diagram of the glass manifold is shown in Figure 1.

3.2 VOST COMPONENTS

The sampling train component parts, except for the glass manifold, are the same as those used for VOST.

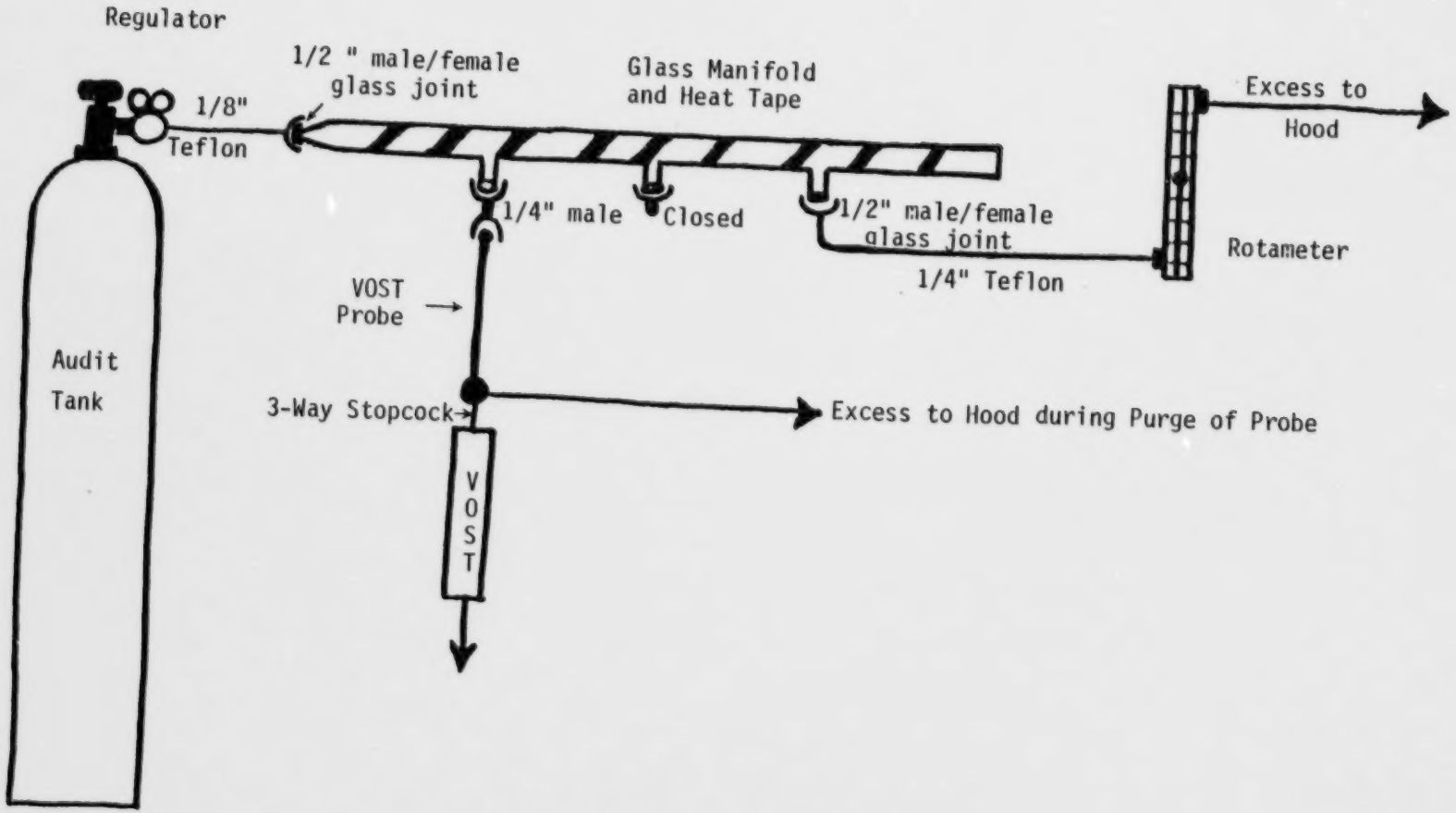

Figure 1. Schematic Diagram of Audit Material Delivery System

3.3 ANALYSIS

Refer to the method specifications and procedures for the VOST method.

4.0 AUDIT MATERIAL (GAS CYLINDERS)

The audit material is supplied in gas cylinders. A maximum of 50 L of gas is to be used for each test run. A two-stage gas regulator is supplied for use with the gas cylinder.

5.0 PROCEDURES

5.1 SAMPLING

5.1.1 Preparation of VOST

All train components should be cleaned and assembled as described in the VOST method used.

5.1.2 Leak-Check Procedure

Before the sampling run, the train is leak-checked as described in the VOST method. During sampling, all connections should be visually examined.

5.1.3 Sample Generation

The gas sample from the audit cylinder is passed through the glass manifold at a flow rate of 2 L/min for 3 minutes to condition the glass system. A glass rotameter is supplied to measure the flow rate at the end of the glass manifold. The VOST probe then should be connected to one port of the glass manifold with 1/4 inch male/female joints and the probe should be purged with audit gas for an additional 2 minutes so that the probe is purged of ambient air before initiation of sample collection. This can be accomplished either by attaching a pump to a three-way stop-cock above the first condenser and drawing audit gas through the probe via the three-way stop-cock or by any other suitable method.

5.2 SAMPLE COLLECTION

After leak-checking and conditioning the glass probe, sample collection is accomplished by opening the valve at the inlet to the first conden-

ser, turning on the pump and sampling at a rate of 0.5 L/min for 20 minutes. The sampling rate is measured with the rotameter which is part of the VOST. The volume of sample for any pair of the traps should not exceed 10 L. The temperature of the gas stream at the inlet to the first sorbent cartridge must be maintained at 20°C during sample collection.

After the collection of 10 L of sample, the train is leak-tested using the procedures described in the method used. If leak-test criteria as described in the method are not met, the samples should not be analyzed. If leak-test criteria are met, the train is returned to atmospheric pressure, the two sorbent cartridges are removed, the end caps are replaced, and analysis of cartridges is performed.

Repeat the sample collection procedure two more times with two new pairs of sample cartridges in the VOST, collect 10 L of sample each time, and perform the analysis.

5.3 SAMPLE ANALYSIS

Follow the procedures described in the method for sample analysis, calibration, and calculations. Report the amount of each constituent in the three samples and the blank values to the Project Officer and the audit supervisor for evaluation.

APPENDIX B
SUMMARY OF SAMPLING AND ANALYSIS PROCEDURES

LABORATORY A

SAMPLING AND ANALYSIS PROCEDURES

I. SAMPLING PROCEDURES

A. VOLATILE ORGANIC SAMPLING TRAIN (VOST)

Equipment: Nutech Model 280/01 B train with several laboratory modifications

Flow rate: 0.5 L/min

Time of collection: 20 min

Total volume collected: Approximately 10 L

B. BAGS

10-L Tedlar bags purchased from Nutech Corp.

II. ANALYSIS PROCEDURES

A. VOLATILE ORGANIC SAMPLING TRAIN (VOST)

Instrumentation: Finnigan 4000 GC/MS/INCOS data system with a Tekmar LSC1 purge and trap apparatus was used for the analysis of VOST cartridges. Tenax and Tenax/charcoal cartridges were simultaneously desorbed and analyzed as a pair.

Column: 8' X 1/8" glass 1% SP-1000 60/80 Carbopack B

B. BAGS

Instrumentation: Varian 3700 gas chromatograph equipped with FID and Nickel-63 ECD. FID was used for the analysis of benzene and vinyl chloride. ECD was used for the analysis of carbon tetrachloride, chloroform, and perchloroethylene.

Column: 8' X 1/8" SS 1% SP-1000 60/80 Carbopack C for FID

8' X 1/8" SS 0.2% Carbowax 1500 on 60/80 Carbopack C for ECD

Sample Loop: 1-cm^3 Teflon loop for ECD
9-cm^3 Teflon loop for FID

Conditions: The GC oven was held at 80°C for 3 minutes, then programmed to 190°C at 10°/minute and held for 10 minutes.

Data Reduction: Electronic integration. The data system used was the Varian Vista 401.

III. CALIBRATION PROCEDURES

GC/MS

All standards except vinyl chloride were prepared in methanol solution in the range of 10 to 100 ppb and injected onto a set of Tenax-Tenax/charcoal cartridges. A vinyl chloride standard was prepared by flowing through the cartridges a known volume of 1.04 ppm vinyl chloride gas standard, which was purchased from Scott Environmental. Bromo-fluoro-benzene was also used as an internal standard.

GC

Bag standards were prepared in the range of 100 to 500 ppb in 10 L of nitrogen by injecting known amounts of each component (pure liquids for all components except vinyl chloride) into the bags and then placing the bag in front of a GC oven at 50°C to enhance vaporization and mixing. Three standards were prepared and analyzed. Dilutions of approximately 10-ppm and 1-ppm vinyl chloride gas standards were made for the calibration of vinyl chloride.

LABORATORY B

SAMPLING AND ANALYSIS PROCEDURES

I. SAMPLING PROCEDURES

A. VOLATILE ORGANIC SAMPLING TRAIN (VOST)

Equipment: Nutech Model 280/01B coupled with laboratory-made components.

Flow rate: 0.5 L/min

Time of collection: 20 min

Total volume collected: Approximately 10 L

B. BAGS

Type: Five-layer bonded bags purchased from Calibrated Instruments Inc. The bag is composed of the following materials, starting with the innermost:

75 µ PE (polyethylene)
40 µ PA (polyamid)
12 µ Al (Aluminum foil)
3-4 µ PVDC (polyvinylide chloride)
12 µ (PETP) (polyester)

II. ANALYSIS PROCEDURES

A. VOLATILE ORGANIC SAMPLING TRAIN (VOST)

Instrumentation: Finnigan 4000 GC/MS with a Tekmar purge and trap apparatus was used for the analysis of VOST cartridges. Tenax and Tenax/charcoal cartridges were desorbed separately and analyzed.

Column: 8' X 1/8" SS 1% SP-1000 on 60/80 Carbopack B

B. BAGS

Instrumentation: Perkin-Elmer gas chromatographs with ECD and PID. ECD was used for analysis of chloroform, carbon tetrachloride and perchloroethylene. PID was used for the analysis of benzene and vinyl chloride.

Columns: 6' X 1/8" glass column 1% SP 1000 Carbopack for ECD

6' X 1/8" glass column 1% SP 1000 on Carbopack B for PID

Sample injection: 500 µL of sample were injected into GC/ECD with gas-tight syringe.

2.5 mL of sample were injected into GC/PID with gas-tight syringe.

Carrier gas: Nitrogen

Conditions: Gas chromatograph with ECD was maintained at 150°C isothermal.
Gas chromatograph with PID was maintained at 70°C for 6 minutes, then programmed to 150°C at 24°C/minute.

Data Reduction: Manually. Peak height measurements were used for GC peaks.

III. CALIBRATION PROCEDURES

GC

Standards were prepared in the range of 100 to 500 ppb in 500 mL glass bulbs containing He or N_2 by injecting known amounts of each component (pure liquids for all components except vinyl chloride). Complete vaporization of the liquid was assumed. At least three standards were prepared and analyzed. Dilutions of 1027-ppm vinyl chloride obtained from Scott were used as calibration standards.

GC/MS

All standards except vinyl chloride were prepared in methanol from pure liquids and injected onto Tenax or Tenax/charcoal cartridges using a flash vaporization technique. Aliquots taken from a gas cylinder, obtained from Scott, containing 1027-ppm vinyl chloride were used as calibration standards to load onto the Tenax-Tenax/charcoal cartridges. The Tenax and Tenax/ charcoal cartridges were loaded for calibration and analyzed separately.

LABORATORY C

SAMPLING AND ANALYSIS PROCEDURES

I. SAMPLING PROCEDURES

A. VOLATILE ORGANIC SAMPLING TRAIN (VOST)

Equipment: Nutech Model 280/01B

Flow rate: 0.5 L/min

Time of collection: 20 min

Total volume collected: Approximately 10 L

II. ANALYSIS PROCEDURES

A. VOLATILE ORGANIC SAMPLING TRAIN (VOST)

Instrumentation: Tracor 560 gas chromatograph with PID and Hall detectors was used for the analysis of VOST cartridges. Tenax and Tenax/charcoal cartridges were desorbed simultaneously using a Tekmar liquid sample concentrator (Model LSC-1) and analyzed. Benzene was analyzed by PID. Chloroform and carbon tetrachloride were analyzed with a Hall detector.

Column: 6' X 1/8" glass column 1% SP 1000 on 60/80 Carbopack for both PID and Hall. Sample passed into PID first and then to a Hall detector.

Carrier gas: Helium, 40 cm^3/min flow

Conditions: GC oven was maintained at 65°C for 5 minutes, then programmed at 220°C at 10°C/minute and held for 10 minutes.

Data Reduction: Electronic integration

I. CALIBRATION PROCEDURES

Standards were prepared in methanol solution in the range of 10 to 100 ppb and injected into a stream of helium being passed into Tenax and Tenax/charcoal cartridges. The loaded cartridges were desorbed and analyzed. Bromofluorobenzene was also used as an internal standard.

LABORATORY D

SAMPLING AND ANALYSIS PROCEDURES

I. SAMPLING PROCEDURES

A. VOLATILE ORGANIC SAMPLING TRAIN (VOST)

Equipment: Nutech Model 280/01B

Flow rate: 0.5 L/min

Time of collection: 20 min

Total volume collected: Approximately 10 L

II. ANALYSIS PROCEDURES

A. VOST

Instrumentation: Finnigan 3300 GC/MS with a Tekmar purge and trap apparatus was used for the analysis of VOST cartridges. Tenax and Tenax/charcoal cartridges were desorbed simultaneously and analyzed as a pair.

Column: 8' X 1/8" glass column 1% SP-1000 on 60/80 Carbopack B.

III. CALIBRATION PROCEDURES

Permeation tubes were used as standards for calibration. Standards were made in the range of 10 to 100 ppb and injected onto Tenax and Tenax/ charcoal cartridges in a stream of nitrogen. The loaded cartridges were desorbed and analyzed by GC/MS.

APPENDIX C
AUDIT RESULTS

TABLE 2. RESULTS OF INTERLABORATORY PRECISION

Activity Audited	Audit Gases	NBS conc. ppb (1)	Lab A Mean + Std. Dev. ppb (3)	Lab B Mean + Std. Dev. ppb (3)	Lab C Mean + Std. Dev. ppb (2) (3)	Lab D Mean + Std. Dev. ppb (3)
VOST (Both sampling & analysis)	carbon tetrachloride	21	10.4±4.7	26.6±3.3	23.0±1.0	18.0±4.2
	chloroform	23	23.1±1.9	29.4±0.5	27.7±2.1	18.5±0.7
	perchloroethylene	29	33.9±3.6	38.8±3.1	NOT AUDITED	32.5±5.0
	vinyl chloride	31	15.5±3.8	10.5±2.6	NOT AUDITED	28.0±5.7
	benzene	18	23.5 ±0.6	23.7±1.0	16.3±1.2	19.0±0.0
VOST Cartridges (analysis only)	carbon tetrachloride	21	12.7±3.2	23.3±1.0	23.7±1.0	19.5±0.7
	chloroform	23	24.3±1.0	26.4±0.9	30.3±6.7	18.5±0.7
	perchloroethylene	29	38.0±0.7	33.4±3.5	NOT AUDITED	34.5±2.1
	vinyl chloride	31	16.4±4.0	14.6±9.7	NOT AUDITED	35.5±5.0
	benzene	18	26.2±1.1	22.5±1.3	21.0±0.0	18.0±1.4

(1) Values obtained by direct GC analysis.

(2) Analysis by GC and not GC/MS.

(3) The laboratory mean and standard deviation were obtained from duplicate or triplicate analysis results.

TABLE 3. RESULTS OF VOST SAMPLING AND ANALYSIS AUDIT (ENTIRE TRAIN)

Audit Gases	NBS (1)	LAB A (11/3/84)						LAB B (11/15/83)						LAB C (3) (11/17/83)						LAB D (12/5/83)					
		Run 1		Run 2		Run 3		Run 1		Run 2		Run 3		Run 1		Run 2		Run 3		Run 1		Run 2		Run 3	
	ppb	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.
carbon tetrachloride	21	5.9	-72	10.1	-52	15.3	-27	24.5	17	24.9	19	30.4	45	24	14	23	10	22	5	15	-29	NO DATA (4)		21	0
chloroform	23	25.2	10	21.7	-6	22.4	-3	28.8	25	29.6	29	29.8	30	30	30	27	17	26	13	18	-22	NO DATA (4)		19	-17
perchloroethylene	29	29.7	2	35.7	23	36.3	25	39.6	37	41.3	42	35.4	22	----------NOT AUDITED----------						29	0	NO DATA (4)		36	24
vinyl chloride	31	18.2	-41	NO DATA (4)		12.8	-59	10.8	-65	7.7	-75	13.0	-58	----------NOT AUDITED----------						32	3	NO DATA (4)		24	-23
benzene	18	24.1	34	22.9	27	23.4	30	22.9	27	24.8	38	23.5	31	17	-6	17	-6	15	-17	19	6	NO DATA (4)		19	6

(1) NBS values were obtained by direct GC analyses.

(2) % accuracy = $\frac{\text{laboratory value - NBS value}}{\text{NBS value}} \times 100$

(3) Analysis by GC and not GC/MS.

(4) Computer malfunction.

TABLE 4. RESULTS OF VOST ANALYSIS AUDIT (CARTRIDGES ONLY)

Audit Gases	NBS (1)	LAB A (11/3/84)						LAB B (11/15/83)						LAB C (3) (11/17/83)						LAB D (12/5/83)					
		Run 1		Run 2		Run 3		Run 1		Run 2		Run 3		Run 1		Run 2		Run 3		Run 1		Run 2		Run 3	
	ppb	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.	ppb	% (2) Acc.
carbon tetrachloride	21	14.9	-29	NO DATA (4)		10.4	-50	NO DATA (5)		22.6	8	24.0	14	22.0	5	22.0	5	27.0	29	20	-5	NO DATA (5)		19	-10
chloroform	23	23.6	3	NO DATA (4)		25.0	9	NO DATA (5)		25.8	12	27.0	17	26.0	13	27.0	17	38.0	61	19	-17	NO DATA (5)		18	-22
perchloroethylene	29	37.5	29	NO DATA (4)		38.5	33	NO DATA (5)		30.9	7	35.8	23	----------NOT AUDITED----------						36	24	NO DATA (5)		33	14
vinyl chloride	31	15.3	-51	13.1	-58	20.8	-33	NO DATA (5)		7.7	-75	21.4	-31	----------NOT AUDITED----------						39	26	NO DATA (5)		32	3
benzene	18	27.0	50	NO DATA (4)		25.4	41	NO DATA (5)		21.5	19	23.4	30	21.0	17.0	21.0	17	NO DATA		17	-6	NO DATA (5)		19	6

(1) NBS values were obtained by direct GC analyses.

(2) % accuracy = $\frac{\text{laboratory value - NBS value}}{\text{NBS value}} \times 100$

(3) Analyses by GC and not GC/MS.

(4) Computer malfunction.

(5) Sample cartridge broken.

TABLE 5. RESULTS OF VOST (ENTIRE TRAIN) VS VOST CARTRIDGES

Activity Audited	Audit Gases	NBS conc. pbs (1)	Lab A Range (ppb)	Lab A Average (4) ppb	Lab A Average (4) % (2) Acc.	Lab B Range (ppb)	Lab B Average (4) ppb	Lab B Average (4) % (2) Acc.	Lab C (3) Range (ppb)	Lab C (3) Average (4) ppb	Lab C (3) Average (4) % (2) Acc.	Lab D Range (ppb)	Lab D Average (4) ppb	Lab D Average (4) % (2) Acc.
VOST (Both sampling & analysis)	carbon tetrachloride	21	5.9-15.3	10.4	-50	24.5-30.4	26.6	27	22.0-24.0	23.0	10	15-21	18.0	-14
	chloroform	23	21.7-25.2	23.1	0	28.8-29.8	29.4	28	26.0-30.0	27.7	20	18-19	18.5	-20
	perchloroethylene	29	29.7-36.3	33.9	17	35.4-41.4	38.8	34	-----NOT AUDITED-----			29-36	32.5	12
	vinyl chloride	31	12.8-18.2	15.5	-50	7.8-13.0	10.5	-66	-----NOT AUDITED-----			24-32	28.0	-10
	benzene	18	22.9-24.1	23.5	31	22.9-24.8	23.7	32	15.0-17.0	16.3	-9	19-19	19.0	6
VOST Cartridges (analysis only)	carbon tetrachloride	21	10.4-14.9	12.7	-40	22.6-24.0	23.3	11	22.0-27.0	23.7	13	19-20	19.5	-7
	chloroform	23	23.6-25.0	24.3	6	25.8-27.0	26.4	15	26.0-38.0	30.3	32	18-19	18.5	-20
	perchloroethylene	29	37.5-38.5	38.0	31	30.9-35.8	33.4	15	-----NOT AUDITED-----			33-36	34.5	19
	vinyl chloride	31	13.1-20.8	16.4	-47	7.7-21.4	14.6	-53	-----NOT AUDITED-----			33-39	35.5	15
	benzene	18	25.4-27.0	26.2	46	21.5-23.4	22.5	25	21.0-21.0	21.0	17	17-19	18.0	0

(1) NBS values were obtained by direct GC analyses.

(2) % accuracy = $\frac{\text{laboratory value - NBS value}}{\text{NBS value}} \times 100$

(3) Analyses by GC and not GC/MS.

(4) Average based on duplicate or triplicate runs.

TABLE 6. BLANK CONCENTRATIONS FROM VOST CARTRIDGES (ppb)

AUDIT GASES	LAB A	LAB B	LAB C	LAB D
carbon tetrachloride	ND	ND	0.1	ND
chloroform	ND	ND	ND	0.8
perchloroethylene	ND	0.1	NOT AUDITED	ND
vinyl chloride	ND	ND	NOT AUDITED	ND
benzene	1.0	0.4	<0.2	0.8

ND = Not detected

TABLE 7. AMOUNTS OF AUDIT GASES FOUND ON TENAX VS TENAX/CHARCOAL CARTRIDGES[1]

Activity Audited	Audit Gases	NBS[2] conc. ppb	Run 1			Run 2			Run 3		
			Tenax ppb	Tenax/ Charcoal ppb	Total ppb	Tenax ppb	Tenax/ Charcoal ppb	Total ppb	Tenax ppb	Tenax/ Charcoal ppb	Total ppb
VOST (Both sampling & analysis)	carbon tetrachloride	21	21.9	2.6	24.5	23.2	1.7	24.9	22.0	8.4	30.4
	chloroform	23	28.4	0.4	28.8	29.1	0.5	29.6	25.4	4.4	29.8
	perchloroethylene	29	39.5	0.1	39.6	41.0	0.4	41.4	34.8	0.6	35.4
	vinyl chloride	31	0.9	9.9	10.8	1.1	6.7	7.8	1.5	11.5	13.0
	benzene	18	22.7	0.2	22.9	24.5	0.3	24.8	22.2	1.3	23.5
VOST Cartridges (analysis only)	carbon tetrachloride	21	20.4	NO DATA[3]	NO DATA[3]	21.3	1.3	22.6	21.5	2.5	24.0
	chloroform	23	27.0	NO DATA[3]	NO DATA[3]	25.5	0.2	25.7	26.8	0.2	27.0
	perchloroethylene	29	32.8	NO DATA[3]	NO DATA[3]	29.6	1.3	30.9	35.7	0.1	35.8
	vinyl chloride	31	1.5	NO DATA[3]	NO DATA[3]	1.2	6.5	7.7	0.4	21.0	21.4
	benzene	18	21.5	NO DATA[3]	NO DATA[3]	21.1	0.4	21.5	23.1	0.3	23.4

(1) Results reported by Laboratory B.

(2) NBS values were obtained by direct GC analyses.

(3) Sample cartridge was broken.

TABLE 8. RESULTS OF BAG SAMPLING AND ANALYSIS AUDIT

Audit Gases	NBS conc. (1) ppb	LAB A (3) (11/3/84) Run 1 ppb	Run 1 % (2) Acc.	Run 2 ppb	Run 2 % (2) Acc.	Run 3 ppb	Run 3 % (2) Acc.	LAB B (3) (11/15/83) Run 1 ppb	Run 1 % (2) Acc.	Run 2 ppb	Run 2 % (2) Acc.	Run 3 ppb	Run 3 % (2) Acc.
carbon tetrachloride	141	230	63	222	57	222	57	121	-14	123	-13	124	-12
chloroform	120	161	34	163	36	160	33	99	-18	102	-15	103	-14
perchloroethylene	126	162	29	153	21	181	44	70	-44	70	-44	73	-42
vinyl chloride	154	126	-18	99	-36	86	-44	101	-34	105	-32	96	-38
benzene	133	147	11	158	19	167	26	90	-32	101	-24	101	-24

(1) NBS values were obtained by direct GC analyses.

(2) % accuracy = $\frac{\text{lab value - NBS value}}{\text{NBS value}} \times 100$

(3) Tedlar bags were used by Lab A. Five layered bags were used by Lab B (See Appendix B).

TABLE 9. RESULTS OF DIRECT ANALYSIS AUDIT

Audit Gases	NBS conc (1) ppb	LAB A (11/3/84) Run 1 ppb	Run 1 % (2) Acc.	Run 2 ppb	Run 2 % (2) Acc.	Run 3 ppb	Run 3 % (2) Acc.	LAB B (11/15/83) Run 1 ppb	Run 1 % (2) Acc.	Run 2 ppb	Run 2 % (2) Acc.	Run 3 ppb	Run 3 % (2) Acc.
carbon tetrachloride	141	238	69	223	58	252	79	135	-4	134	-5	131	-7
chloroform	120	176	47	169	41	174	45	117	-3	115	-4	115	-4
perchloroethylene	126	104	-17	102	-19	97	-23	126 (3)	-0	120	-5	116	-8
vinyl chloride	154	87	-44	131	-15	136	-12	87 (3)	-44	107	-31	101	-34
benzene	133	160	20	179	35	180	35	88	-34	117	-12	119	-11

(1) All values were obtained by direct GC analyses.

(2) % accuracy = $\frac{\text{laboratory value - NBS value}}{\text{NBS Value}} \times 100$

(3) According to Laboratory B, these values were the result of a bad injection; thus, they were not used in calculation of the average.

TABLE 10. RESULTS OF DIRECT ANALYSIS VS. BAG ANALYSIS

Activity Audited	Audit Gases	NBS conc. ppb (1)	Lab A (4) Range (ppb)	Lab A (4) Average (3) ppb	Lab A (4) Average (3) (2)% Acc.	Lab B (4) Range (ppb)	Lab B (4) Average (3) ppb	Lab B (4) Average (3) %(2) Acc.
BAG (Both sampling & analysis)	carbon tetrachloride	141	222-230	225	60	121-124	123	-13
	chloroform	120	160-163	161	34	99-103	101	-16
	perchloroethylene	126	153-181	165	31	70-73	71	-44
	vinyl chloride	154	86-126	104	-33	96-105	101	-34
	benzene	133	147-167	157	18	90-101	97	-27
DIRECT GAS Analysis by GC only	carbon tetrachloride	141	223-252	238	69	131-135	133	-6
	chloroform	120	169-176	173	44	115-117	116	-3
	perchloroethylene	126	97-104	101	-20	116-126	121	-4
	vinyl chloride	154	87-136	118	-23	101-107	104	-33
	benzene	133	160-180	173	30	117-119	118	-11

(1) NBS values were obtained by direct GC analysis.

(2) % accuracy = $\frac{\text{laboratory value - NBS value}}{\text{NBS value}} \times 100$

(3) Average based on triplicate runs.

(4) Tedlar bags were used by Lab A. Five layered bags were used by Lab B (See Appendix B)